LIBBY #3

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QUALITY ASSURANCE PROJECT PLAN

for
Libby, Montana

PERFORMANCE EVALUATION STUDY FOR ANALYTICAL METHODS FOR ASBESTOS IN SOIL

PART B
REVISION 1

PREPARATION OF PERFORMANCE EVALUATION SAMPLES

January 24, 2003

Redacted

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APPROVAL PAGE

This Quality Assurance Project Plan for the Libby Montana Performance Evaluation Study - Part B has been prepared by the U.S. Environmental Protection Agency, Region 8, with technical support from Syracuse Research Corporation. Study activities addressed in this Plan are approved without condition.

Program Approval
Jim Christiansen
USEPA Remedial Project Manager
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Date 3/25/03

Technical Approval
Mary Goldade
USEPA Regional Chemist

Date 3/25/03

DOCUMENT REVISION HISTORY

Revision Number	Date	Reason for Revision
0	11/13/01	Not Applicable
1	3/25/03	Incorporate experience gained from pilot studies

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LIST OF ABBREVIATIONS AND UNITS

CAR	Corrective Action Request
g	gram
GLP	Good Laboratory Practices
HEPA	High Efficiency Particulate Air
IR	Infrared Spectrometry
ISO	International Standards Organization
kg	kilogram
mL	milliliter
mm	millimeter
PE	performance evaluation
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
R/PE	Reference/Performance Evaluation
SEM	Scanning Electron Microscopy
SOP	Standard Operating Procedure
TEM	Transmission electron microscope
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
XRD	X-ray Diffraction
°C	degrees Celsius
"	inch

A. PROJECT MANAGEMENT

A4. PROJECT/TASK ORGANIZATION

The project is being planned by the U.S. Environmental Protection Agency (USEPA) Region 8. The following individuals are the USEPA project personnel with overall responsibility for the design and conduct of this project:

- Jim Christiansen, USEPA Remedial Project Manager (RPM), oversees all remedial activities at the Libby Operable Unit 4 site and is the primary data user and decision-maker at the site.

- Mary Goldade, USEPA Regional Chemist, will serve as lead investigator for USEPA and will be responsible for the technical aspects of the study.

Work to be conducted under Part B of this Quality Assurance Project Plan (QAPP) will be performed primarily by the U. S. Geological Survey (USGS) in Lakewood, Colorado, working under an Inter-Agency Agreement with the USEPA Region 8. The project leader for the USGS is Steve Wilson, PhD, who has helped develop this project plan and will provide senior technical input and oversight of all USGS activities.

A5. PROBLEM DEFINITION AND BACKGROUND

Site Description

Libby, Montana is a community located near an open pit vermiculite mine which began limited operations in the 1920's and was operated on a larger scale by W.R. Grace Company from 1963 to 1990. Vermiculite from this mine contains varying amounts of amphibole asbestos. As a consequence, soils in and about the community of Libby may have become contaminated with mine-related asbestos, either as a result of placement of bulk material (vermiculite, mine waste, ore, etc) that contains asbestos, or from historic aerial deposition of airborne fibers released by past mining and milling operations. Asbestos in area soils could be serving as an on-going source of asbestos release into air. Thus, there is a vital need for EPA to be able to analyze samples of soil and soil-like material from the community in order to measure the asbestos content and assess the potential risk to area residents.

Need for Improved Analytical Methods

While there are a number of analytical methods currently available for the analysis of asbestos in solid media, these have predominantly been developed to support analysis of various building materials that contain relatively high asbestos contents, and the methods are not generally well-suited for assessing lower-level asbestos contamination (a few percent or less) in environmental media such as soil. In addition, some of these methods are relatively slow and costly. For this reason, the USEPA is currently working to develop and refine methods that will be suitable for the analysis and characterization of relatively low levels (e.g., 0.05-5%) of asbestos in soil and other related media, and that are relatively fast and cost efficient. This type of method is needed to support risk management decision-making regarding the need for remediation of potential source materials at the site.

Need for Reference Material and Performance Evaluation Samples

A key part of the approach for evaluating the performance of different analytical methods is the availability of a set of reference materials that contain known amounts of asbestos in soil. These may be used either as reference (calibration) materials for the different methods, or may be submitted as blind performance evaluation samples to help characterize the accuracy, precision, and sensitivity of a particular method. Ideally, such reference or performance evaluation (R/PE) materials are prepared using site-specific matrix (soil) and site-specific asbestos material. At present, such site-specific R/PE materials do not exist.

Phased Approach

The overall project (referred to as the Libby PE project) is being performed in three parts:

<u>Part A</u> is the collection of the raw materials (Libby soil, Libby asbestos) needed to prepare the Libby site-specific R/PE samples.

<u>Part B</u> is the preparation, characterization and mixing of site-specific soil with asbestos fibers or particles to generate a series of site-specific R/PE samples.

<u>Part C</u> is the distribution of the reference materials to multiple laboratories for use in method calibration, followed by round-robin testing of the PE samples to establish the performance attributes and to estimate initial acceptance criteria for each analytical method.

Part A has been completed as described below. The document describes Part B of the project (preparation of R/PE materials). The project plan for Part C of the document will be presented separately. In addition, several pilot-scale studies were performed in order to help optimize the overall approach. These are summarized below.

Summary of Pilot Scale Investigations

Originally (see USEPA 2001), it was envisioned that the R/PE materials that would be required to support this project would include the following:

Standard 1 - Amphibole Fibers in Synthetic Matrix (Ground Quartz)
Standard 2 - Amphibole Clumps in Synthetic Matrix (Ground Quartz)
Standard 3 - Amphibole Fibers in Libby Site Soils
Standard 4 - Amphibole Clumps in Libby Site Soils
Standard 5 - Chrysotile Fibers in Synthetic Matrix (Ground Quartz)
Standard 6 - Chrysotile Fibers in Libby Site Soils

Ground quartz was selected as the initial test matrix because it was expected to provide little or no interference with most of the analytical methods being contemplated. An initial pilot-study was performed in which several different concentrations of Libby amphibole fiber were added to and mixed with ground quartz particles. The details of this pilot study are presented in Technical Memo 4 (USEPA 2002a). These quartz-based materials were analyzed by two laboratories using scanning electron microscopy (SEM) and one laboratory using infra-red reflectance spectroscopy (IR). As discussed in USEPA (2002a), the results of this pilot study suggested that the asbestos fibers added to quartz particles tended to adhere to the quartz grains (probably due to electrostatic forces), and the appearance of the material was dissimilar to soil samples from the site. On this basis, it was concluded that the results of any analyses of this class of R/PE material would probably not provide much insight into how the methods would perform using site soils, and the use of quartz-based R/PE materials has not been pursued further.

For this reason, attention shifted to pilot-scale testing of soil-based samples. The first soil-based test materials were prepared by adding Libby amphibole fibers to two different soil matrices: one soil collected from Libby, and one soil collected from the grounds of the Denver Federal Center. These materials were referred to as Interim Soil Test Materials (ISTMs). The details of this pilot study are presented in Technical Memo 5 (USEPA 2002b). In brief, the soils were dried, disaggregated, and sieved through a 2 mm screen. Asbestos fibers were added as a slurry and mixed, and the resulting mixture was dried and disaggregated. As before, these samples were analyzed by SEM and IR. The results of this pilot study indicated that even though neither of the methods (SEM or IR) for analysis of asbestos in soil had high accuracy, both offered the potential of being useful as semi-quantitative screening tools for assessment of soil samples. An important finding was that even though the soil matrix had been sieved to a 2 mm screen size, the laboratories involved in the ISTM1 studies reported that particle size heterogeneity in the ISTM1 samples caused difficulty in the analysis.

This concern (analytical difficulty due to particle size heterogeneity) was originally considered to be a "necessary evil" because asbestos particle size is an important factor that influences asbestos toxicity. That is, grinding samples to reduce particle size heterogenity was originally considered to be inappropriate because grinding would alter the asbestos particle size distribution and hence preclude the ability to consider asbestos particle size in the hazard assessment.

However, considering the analytical difficulties faced by the laboratories, and recognizing that risk management decisions could be based on total asbestos content (used as a metric that is expected to be correlated with current or potential future risk), it was decided that grinding soil samples could be performed.. Consequently, the next series of pilot studies involved preparation of a new set of Interim Soil Test Materials (this set being referred to as ISTM2s) that were ground before analysis. The details of the ISTM2 preparation are presented in Technical Memo 9 (USEPA 2003). In brief, the soil matrix was initially ground to a particle size of about 1 mm before spiking with Libby amphibole fibers. As before, spiking and mixing occurred as a aqueous slurry. After drying of the spiked materials, the samples were was re-ground to a 60-mesh (250 um) size and distributed to bottles. These materials were submitted to 5 different laboratories for analysis by one or more of several different methods, including IR, PLM, SEM, and TEM, in accord with draft SOPs developed to support this phase of the pilot project.

The results of the ISTM2 analyses are detailed in USEPA (2003). In brief, most methods showed reasonable promise in the detection and quantification of Libby asbestos in site soils, with moderate to good accuracy and precision, and detection limits extending below 1% in several cases.

Based on this indication of potential success, it was decided to cease pilot-scale studies and proceed to the development of R/PE materials to support the full PE project. In addition to the R/PE samples, consensus was reached that the study would also benefit from preparation of a second type of samples, referred to as Quality Assurance (QA) samples. These samples will be authentic Libby field soil samples that contain asbestos from site-related sources but are not spiked with any added asbestos. These samples have the benefit of being entirely authentic, and may better capture the variability in soil type and potential interferences that may exit at different locations across the community than the R/PE samples. Analysis of this type of soil allows for a direct evaluation of within and between-laboratory precision, and will also allow for an evaluation of accuracy once a consensus value is established.

A6. PROJECT/TASK DESCRIPTION

The plan for preparing R/PE samples of site-specific asbestos in site-specific soil matrix consists of four steps:

1) Collect and prepare the site specific soil to use in making the R/PE samples

2) Prepare the asbestos material to be used in preparing the R/PE samples

3) Mix a known mass of soil matrix with a known mass of asbestos to produce a range of known concentrations (expressed as mass percent)

4) Prepare aliquots of the R/PE samples suitable for distribution to analytical laboratories for use as reference materials or as blind PE samples

The plan for preparing QA samples of site-specific soils consists of 3 steps:

1) Identify on-site locations where soil is contaminated with site-related asbestos and where sufficient sample mass may be collected to prepare an adequate number of QA samples.

2) Collect and prepare a series of QA samples.

3) Prepare aliquots of the QA samples suitable for distribution to analytical laboratories in a random and blind fashion

A7. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

Because Part B of the project is intended only to prepare the R/PE and QA samples, no quantitative measurements that require Data Quality Objectives are required.

B. MEASUREMENT DATA ACQUISITION

Measurement data (the results of the analysis of R/PE and QA samples by a variety of analytical techniques) will be obtained in Part C of this project. The following sections describe how the R/PE and QA samples will be prepared.

B1. R/PE SAMPLE PREPARATION PROCESS DESIGN

Step 1. Collect and Prepare the Soil Matrix to Be Used in Making the R/PE Samples

Soil Source

Results of the ISTM2 pilot study indicated that the success of some analytical methods for asbestos in soil may depend in part on the attributes of the soil matrix used to prepare the sample. Although data from USGS indicate that most soils in Libby have very similar mineral content, soils do vary somewhat in color and texture, and there are variations in the minor mineral components (USEPA 2002c). Inspection of a series of authentic Libby field soil samples by USGS staff indicated that most samples could be classified as "brown", "tan", or "rocky". Samples classified as "rocky" were judged to be similar to road base (e.g., from a driveway or roadway). Because the chief focus of the project is on evaluation of potential contamination in residential yards, it was decided that R/PE samples would be prepared using both "brown" and "tan" soil matrix, but that "rocky" (road base) matrix would not be utilized at present.

Site-specific sampling results were used to identify multiple locations in Libby where "brown" and "tan" soil that contained little or no asbestos could be collected. These results are presented in Tech Memo 6 (USEPA 2002c). In accord with this memo, "brown soil" matrix material has been collected from two locations in Libby, identified as PE-00005, PE-00013, PE-00018, and PE-00019. "Tan soil" matrix material was also been collected from two locations in Libby, identified as PE-00002, PE-00012, and PE-00015. All of these samples were collected from locations where previous samples were found to be below detection limits for asbestos by PLM and below detection limits for amphibole content by XRD (X-ray Diffraction).

Confirmation that the Samples are "Clean"

Prior to preparing any reference materials or PE samples with these soil matrices, representative samples from each bucket will be re-analyzed by PLM, XRD, and by SEM. Assuming that no evidence of significant asbestos contamination is detected by any method, these materials will be prepared as follows.

Soil Matrix Preparation

1. Dry all samples

2. Composite the buckets of "brown" soil
   Composite the buckets of "tan" soil

3. Coarse-sieve each composited soil sample to remove any material larger than 1/4 inch.

4. Grind the remaining soil (< 1/4 inch) to a particle size of about 1 mm. Mix well.

The total mass that is desirable for each soil matrix (dried, coarse-sieved, and ground) is approximately 50 kg.

Step 2. Prepare the Asbestos Spiking Material

Asbestos material has been collected from 30 different locations within the Libby mine site by USGS and EPA scientists. These raw starting materials are in the form of lumps of ore (vein rock) that are enriched in amphibole asbestos. For the purposes of this project, all of these samples are considered to be representative of the Libby mine.

Selection of Starting Material

After visual and spectroscopic analysis of the 30 ore samples, six samples have been identified as being relatively pure (60-90% asbestos). These six samples will be used to generate the supply of amphibole spiking material for this project.

Crushing of amphibole samples

Each of the six samples have been crushed at the USGS laboratory using a 1 ton hydraulic press located in a HEPA hood. Large chunks of vein material (2-10 cm^3) were double-wrapped in plastic and cloth, then inserted between the steel plates of the press. The press opening was gradually reduced until the sample volume was less than 1 cm^3. Material from each site was processed individually. After all the sample from one site was processed, the press was vacuumed and then wiped clean.

The crushed samples were doubled bagged in Ziplock bags and then placed in individual 5 gallon containers. The samples (estimated weight 30 pounds) was processed as described below in a laboratory with a HEPA hood to allow safe handling of the fibrous material.

Drying of vein material

The ore material was placed in a HEPA hood and spread out to a depth of approximately 2-4 cm. in 14" x 18"x 1" plastic trays lined with aluminum foil. The trays were positioned under a set of two heat lamps and dried for a minimum of 24 hours until visual inspection of the material indicates that it was dry and ready for further processing.

Coarse Grinding

Dried vein material prepared as described above was processed using a Quaker City Mill, Model 4-E horizontal grinder equipped with a 1 inch diameter steel auger and 4 inch steel grinding plates. Sample grinding was performed in the HEPA hood. Grinding plates were adjusted so that a maximum particle size of approximately 0.2 cm^3 is achieved. All material were collected in plastic bags positioned at the exit port of the mill.

Mixing and Splitting of Coarse-Ground Material

Working in batches, asbestos material prepared as described above was transferred to a 20 gallon plastic lined steel drum which is fitted with a USGS-designed steel mixing baffle. After all the ground sample was added to the barrel, the barrel was carefully sealed and all outside surfaces of the barrel were washed down and dried to insure minimal contamination of the working area. The barrel was moved to a horizontal roller mill located outside the HEPA hood and mixed for a period of 2-4 hours. After mixing, the barrel was transferred to the HEPA hood, the mixing baffle removed. Aliquots of the asbestos material were then removed as needed..

Step 3. Preparation of Reference Materials and PE Samples

Reference materials and PE samples will be prepared by mixing measured masses of soil with measured masses of spiking material (prepared as described in Step 2, above). Thus, all concentrations are expressed in units of mass percent. Table 1 lists the target concentrations of asbestos in the Reference Materials and the PE samples. Not all of the materials in Table 1 are required simultaneously. Rather, studies may proceed with only a subset of samples, so long as the subset provides good coverage of the concentration range of concern for a particular method.

All blending of soil matrix and spiking material will be performed as an aqueous slurry. The slurry mixture will be transferred into a mixing container and allowed to mix for 2-4 hours. After mixing, the sample will be removed from the container and spread out on drying trays, positioned on top of 12" x 24" laboratory hot plates located in the HEPA hood. The hot plates will be set to 30°C and the sample allowed to dry overnight. Once dried, the material will be re-mixed in a roller mill for 1-2 hours.

After drying and re-mixing, the entire batch of each material will be ground by passing through a vertical plate grinder set to produce a maximum soil particle of about 250 um (60 mesh), as described in SOP ISSI-LIBBY-01.

Step 4. Preparation and Labeling of Sample Bottles

After grinding, all reference materials and PE samples will be distributed into bottles using a spinning riffle splitter located in the HEPA hood. The bottles will be of a size that the sample mass in the bottle fills the bottle approximately half-way. An aliquot of material will be loaded into the sample reservoir and once the bottling ring is set in motion, sample will be allowed to flow onto the splitting ring. The flow of material will continue until all the material is dispersed from the delivery reservoir and evenly distributed between all the bottles in the ring. At the end of the bottling period, each bottle will be wiped with cleaning cloths to remove any sample located on the outside of the container. If necessary, a plexiglass shield will situated in front of the delivery chute so as to minimize the flow of high velocity air across the bottle ring. This will minimize the loss of fibers to the atmosphere and maximize the amount of fibrous material delivered to the bottle.

Bottles of reference material will contain about 50 grams each, and will be labeled as follows:

LIBBY PE STUDY REFERENCE MATERIAL
Nominal Asbestos Concentration = x% by Weight

Bottles of PE samples will contain about 20 grams each, and will be labeled as follows:

LIBBY PE STUDY PERFORMANCE EVALUATION SAMPLE
Sample Number = PE-xxxxx

USGS will track blind PE sample IDs and provide the nominal concentration information for each sample ID assignment. Each bottle will be placed and sealed inside a plastic bag before shipment in order to guard against breakage and contamination during shipment.

B2. QC SAMPLE PREPARATION PROCESS DESIGN

All QC samples will be authentic Libby field soil samples that are prepared for analysis by drying, coarse sieving and grinding in accord with SOP ISSI-LIBBY-01.

QC samples will be prepared in relatively large lots (10-20 kg each). The first QC sample will be selected to contain an asbestos level in the range of about 1-2%. After the available supply of that material is approximately 1/2 exhausted, the second QC sample will be prepared. Each subsequent sample will be selected to represent a different combination of soil matrix type and asbestos content (spanning the range from ND to about 5% by weight). Tech Memo 6 (USEPA 2003c) describes the process used to select sampling locations where authentic Libby filed soils can be collected to achieve this range of asbestos concentrations and soil types.

QC samples will be prepared in bottles of about 20 grams each, and will be submitted to laboratories in a blind and random fashion and will be labeled and tracked similarly as PE samples.

B3. SAMPLE IDENTIFICATION, DOCUMENTATION, CUSTODY, HANDLING, AND ARCHIVE

The data needed to translate between coded sample number on bottles of QC and PE material and the sample identity (i.e., nominal content of asbestos) will be maintained in a database by USGS in order to decode analytical results. All QA and PE samples will be provided to laboratories in a random and blind fashion.

<u>Sample Handling and Archive</u>

All R/PE samples prepared for this program must be retained by USGS or by USEPA in a dry, secure (locked with limited access) room-temperature storage facility. Samples must be stored in an organized manner such that quick retrieval is possible. All samples will be held under chain-of-custody in accord with the basic principles described in CDM SOP 1-2, until the Field Project Leader (as directed by the RPM) indicates that these samples may be shipped to a participating laboratory for analysis.

<u>Sample Shipment</u>

Shipment of samples to participating laboratories will be performed under chain of custody in accord with CDM SOP 1-2. All shipments will follow standard laboratory practices except that **NO VERMICULITE** may be used in packing the materials.

Decontamination

Decontamination is defined as physically removing contaminants and foreign material (e.g., dust, oil, detergent) or altering their chemical character to nonreactive/inert substances. All laboratory equipment and vessels used in the preparation of these R/PE and QA samples must be decontaminated using standard good laboratory practices prior to use and between repeated uses to minimize the potential for cross-contamination of samples.

C. ASSESSMENT AND OVERSIGHT

C1. ASSESSMENTS AND RESPONSE ACTIONS

Audits

Assessment of the preparation of R/PE and QC samples may be conducted through oversight of activities or audits. The purpose of the oversight (audit) activities will be to document preparation procedures, to determine if activities are proceeding in accord with project requirements and to document any changes, additions or deletions that have occurred during field sampling and analysis and to identify and immediately implement any corrective actions.

Audits will evaluate procedures to ensure that they follow Good Laboratory Practices (GLP) Guidelines and to ensure that they do not conflict with project requirements. If conflicts are noted, these must be addressed so that project requirements are met.

If audits reveal that project requirements are not met, then corrective action for the deviation must be requested, reviewed and reported. Results for all audits must be documented and submitted to the USEPA Remedial Project Manager. Information in the audit report should include:

- Type and date of audit
- Summary of procedures reviewed
- Results of the review/audit, including any non-conformances noted
- Corrective Action Request(s) [CAR], if non-conformance noted

- Date by which CAR must be received with response

If a CAR is required, a follow-up audit must be performed upon receipt of the CAR to ensure that corrective actions were implemented. A follow-up audit report describing the new findings must be submitted to the USEPA Remedial Project Manager. More detailed information regarding corrective action procedures is provided in the next section.

Corrective Action Procedures

Two types of corrective actions may result from audits and/or oversight: immediate and long-term. Immediate corrective actions include correcting deficiencies or errors or correcting inadequate procedures. Long-term corrective actions are designed to eliminate the sources of deficiencies or errors. If either type of corrective action is deemed necessary following an audit, each step in the following procedures must be documented:

- Identify the deviation
- Request a corrective action
- Report the problem the USEPA Remedial Project Manager
- Review the corrective action response
- Perform a follow-up audit to ensure the deviation is not recurring

C2. REPORTS TO MANAGEMENT

All original documentation associated with preparation of the R/PE samples and the QA samples will be retained by the USGS or by USEPA. Originals will be available for review and reproduction will be provided upon request. All records and logbooks will be maintained until written directions for their disposal. Raw data will include but may not be limited to sample preparation logsheets, data collection logsheets, and supporting calculations.

D. DATA VALIDATION AND USABILITY

Not applicable.

E. REFERENCES

USEPA. 2001. Project Plan for the Performance Evaluation Study for Analytical Methods for Asbestos in Soil, Part B (Revision 0). Preparation of Performance Evaluation Samples

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USEPA. 2002a. Technical Memo 4. Summary and Evaluation of Interim Quartz Test Material Sample Analysis by SEM and IR. Prepared by US Environmental Protection Agency Region 8 with Technical assistance from Syracuse Research Corporation.

USEPA. 2002b. Technical Memo 5. Summary and Evaluation of Interim Soil Test Material Sample Analysis by SEM and IR. Prepared by US Environmental Protection Agency Region 8 with Technical assistance from Syracuse Research Corporation.

USEPA. 2002c. Technical Memo 6. Collection of Bulk Libby Field Soil Samples for Use in the Asbestos in Soil Performance Evaluation (PE) Study. Prepared by US Environmental Protection Agency Region 8 with Technical assistance from Syracuse Research Corporation. 11/04/02

USEPA. 2003. Technical Memorandum 9. Summary and Evaluation of Interim Soil Test Material (Set 2) Analysis by IR, PLM, SEM and TEM. Prepared by US Environmental Protection Agency Region 8 with Technical Assistance from Syracuse Research Corporation.